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10/30/97

26914 U.S. PTO  
60/064284

Subclass	Class
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ISSUE CLASSIFICATION  
**SCANNED**



60/064284  
10-30-97

PROVISIONAL  
APPLICATION  
NUMBER

SERIAL NUMBER 60/064,284 PROVISIONAL	FILING DATE 10/30/97	CLASS	SUBCLASS	GROUP ART UNIT	EXAMINER
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APPLICANTS DENNIS H. GREEN, ARVADA, CO; JEFF MUELLER, BOULDER, CO.

\*\*CONTINUING DATA\*\*\*\*\*  
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\*\*FOREIGN APPLICATIONS\*\*\*\*\*  
VERIFIED

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Foreign priority claimed 35 USC 119 conditions met	<input type="checkbox"/> yes <input type="checkbox"/> no <input type="checkbox"/> yes <input type="checkbox"/> no	AS FILED →	STATE OR COUNTRY CO	SHEETS DRWGS. 1	TOTAL CLAIMS	INDEP. CLAIMS	FILING FEE RECEIVED \$75.00	ATTORNEY'S DOCKET NO. 3376-19PROV
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Verified and Acknowledged  
Examiner's Initials  
DOUGLAS W SWARTZ  
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DENVER CO 80203

TITLE METHOD OF REMOVING SUSPENDED AND COLLOIDAL SOLIDS FROM COPPER  
LEACHING SOLUTION

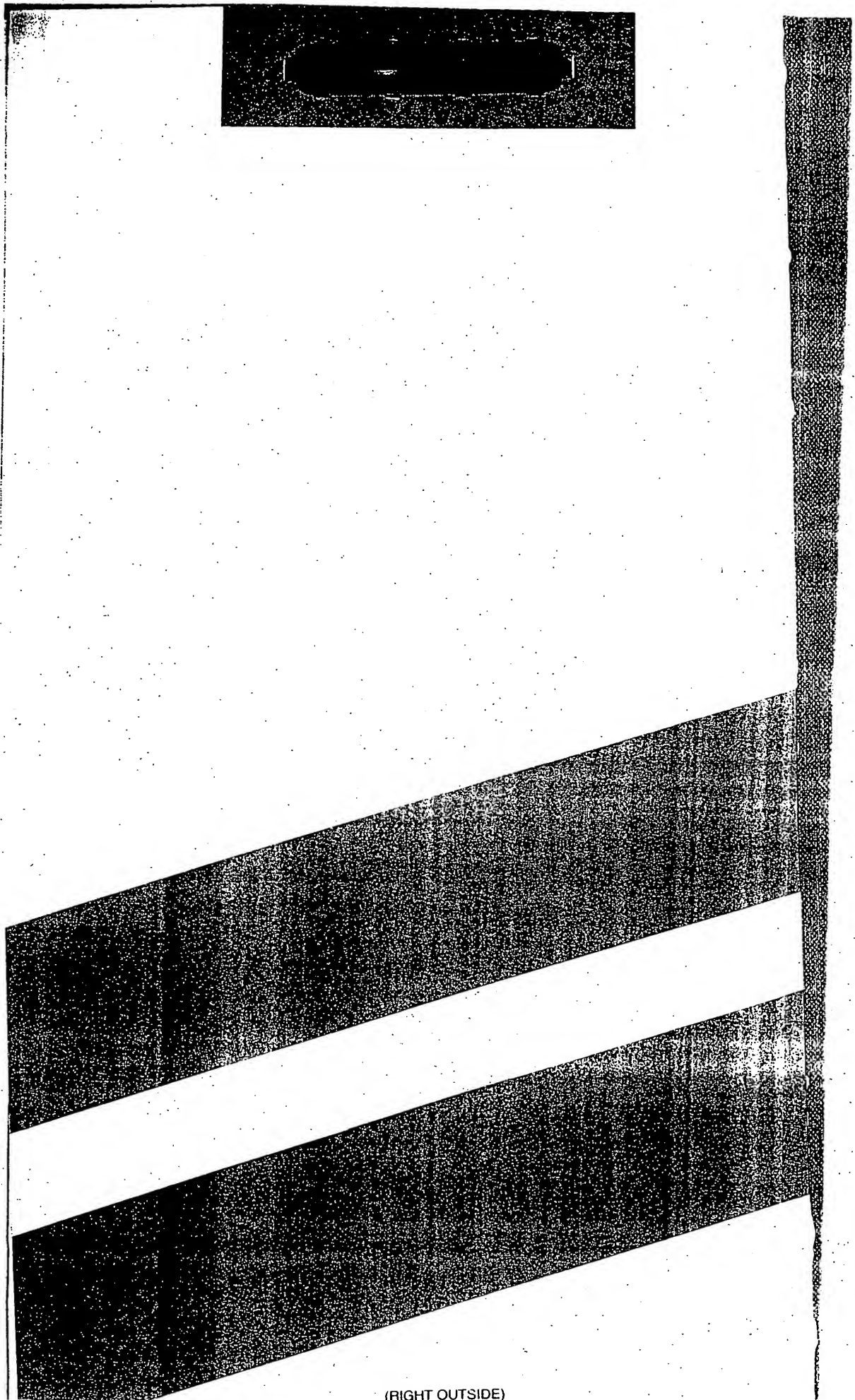
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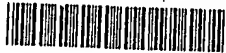
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60/064284



10/30/97

# PATENT APPLICATION



60064284

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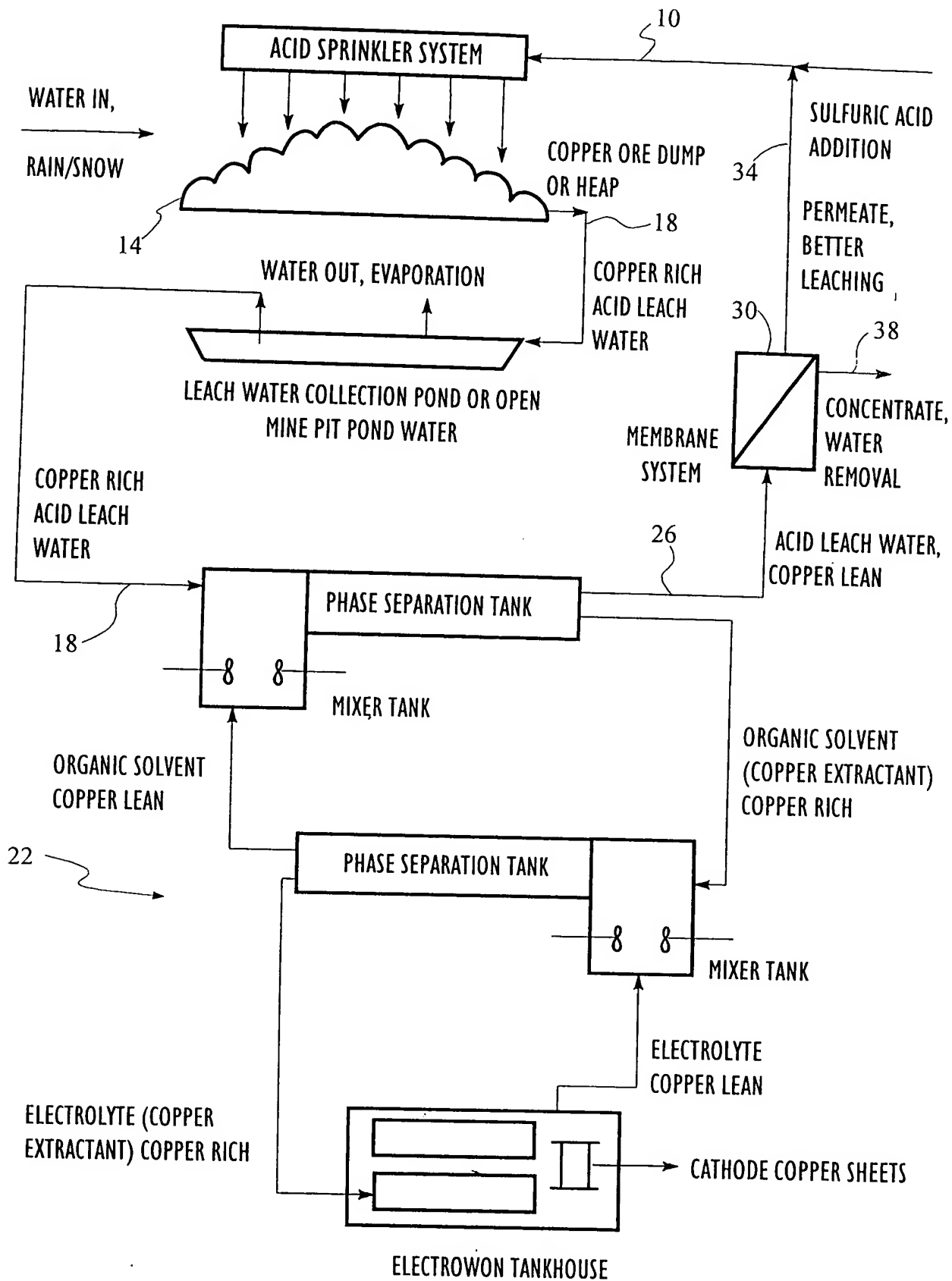


FIG. 1

26914 U.S. PTO

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PATENT APPLICATION SERIAL NO. \_\_\_\_\_

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# PROVISIONAL APPLICATION COVER SHEET

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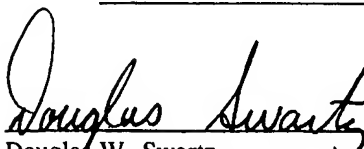
a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(b)(2).

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INVENTOR(s)/APPLICANT(s)					
LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)		
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TITLE OF THE INVENTION (280 characters max)					
"METHOD OF REMOVING SUSPENDED AND COLLOIDAL SOLIDS FROM COPPER LEACHING SOLUTION"					
CORRESPONDENCE ADDRESS					
Douglas W. Swartz Sheridan Ross P.C. 1700 Lincoln Street, Suite 3500 Denver					
STATE	Colorado	ZIP CODE	80203	COUNTRY	United States of America
ENCLOSED APPLICATION PARTS (check all that apply)					
X	Specification	Number of Pages	20	x	Small Entity Statement
X	Drawing(s)	Number of Sheets	1		Other (specify)
METHOD OF PAYMENT (check all that apply)					
X	A check or money order is enclosed to cover the filing fees			PROVISIONAL FILING FEE AMOUNT (\$)	\$75.00
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The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

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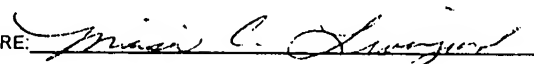
  
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# METHOD OF REMOVING SUSPENDED AND COLLOIDAL SOLIDS FROM COPPER LEACHING SOLUTION

## FIELD OF THE INVENTION

The present invention relates generally to a leaching process and specifically to a leaching process in which the pregnant leach solution is subjected to ultra or microfiltration.

### I. THE PROBLEM

The present invention generally relates to the removal of copper ions from copper ore, and more particularly to enhanced removal of copper ions using a microfiltration or ultrafiltration membrane process to remove suspended solids from copper pregnant leach solution. This process improves the copper recovery process through more efficient copper extraction and lower operating costs.

The techniques used to remove copper from raw ore determine the overall efficiency of the copper mining operation. Hydrometallurgical copper mining operations using a leaching system and a copper extraction plant, such as a solvent extraction/electrowinning (SX/EW) plant, are

now accepted processes in the copper mining industry. Currently, electrowon copper accounts for about 30% of total U.S. copper production. Worldwide, there are more than 26 major heap, dump, or in-situ leaching operations using SX/EW, with a total capacity of -800,000 tons of copper annually. The industry trend continues towards this technology as higher-grade ores are depleted and smelting costs increase. Other advantages of this technology, such as the ability to process low-grade ores, low labor requirements, ease of operation in remote areas, and low operating costs, make it attractive to mining companies.

"Copper hydrometallurgy", in which copper ions are reached or otherwise extracted from raw ore using liquid chemical agents, has been of interest since as early as the 17<sup>th</sup> century when copper recovery methods involving iron precipitating agents from sulfuric acid based copper solutions were tested. The hydrometallurgical circuit consists of copper leaching and copper recovery.

First, a copper leaching agent, "lixiviant", is selected for use in leaching copper ions from copper ore. Representative lixiviants include but are not limited to

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sulfuric acid ( $\text{H}_2\text{SO}_4$ ), a combination of  $\text{H}_2\text{SO}_4$  and ferric sulfate,  $\text{Fe}_2(\text{SO}_4)_3$  (primarily for sulfide containing ore materials), acidic chloride solutions (e.g. ferric chloride,  $\text{FeCl}_3$ ) or cupric chloride,  $\text{CuCl}_2$ ), nitrate solutions, ammonia, and ammonium salt compositions. Sulfuric acid is by far the most common lixiviant. The lixiviant is applied to the ore (which is stacked or piled in a large heap or dump) via a sprinkler type system and allowed to percolate downwardly into the ore. As a result, copper ions are leached from the ore and collected within the lixiviant to generate a lixiviant product that consists of a copper ion concentration (also known as "pregnant leach solution"). The lixiviant exits the bottom of the ore and is collected. Further information regarding the lixiviant leaching process is disclosed in U.S. Patent No. 5,476,591 to Green et al., which is incorporated herein by reference.

Next, a copper recovery process is used to selectively extract copper from the collected lixiviant. Representative copper recovery processes include but are not limited to solvent extraction/electrowin (SX/EW), direct electrowinning, ion exchange - electrowin (IX/EW), and iron

precipitation. Solvent extraction/electrowin is presently the most common copper recovery process. SX/EW technology was implemented in the 1960's with the development of special organic extractants for copper. The SX/EW process consists of three closed solution loops. In the first loop, the acid leach solution containing valuable copper ions and a multitude of other metal ions is fed into a mixer/settler tank where it is contacted with a copper-extracting organic liquid, commonly referred to as "lix". The "lix" preferentially extracts from 70 to 90% of the copper ions from the acid leaching feed solution. The second closed loop extraction step involves contacting the loaded organic \with an electrolyte stream from the electrowinning process. The copper ions are transferred from the organic solution or "lix" to the lean electrolyte. In the third and final closed loop, the rich electrolyte flows between a cathode plate and an insoluble anode, where 70 - 90% of the copper is removed through 'plating." The electrochemical cell "plates" a stainless steel electrode with copper using an applied current. The copper plated cathode plates are then

periodically removed from the process to obtain the solid, high purity copper product.

The copper leaching - copper recovery process must be improved to overcome inherent problems such as copper extraction inefficiencies due to the presence of suspended solids. Suspended solids and colloidal solids enter the copper extraction process (typically SX/EW) with the pregnant leach solution 18, and remain and buildup in the copper extraction process. Typical suspended solids concentrations in the pregnant leach solution 18 range from 1-50 mg/L. During rain or storm events when the leach solution collection reservoir bottoms are stirred-up and rain carries suspended solids into the leach solution collection reservoirs, suspended solids levels can double or triple for a short period of time (1 -3 days).

The suspended solids which enter the copper recovery process with the pregnant leach solution 18 have adverse effects on the copper recovery process. For example, suspended solids can plug copper ion exchange resin beds used in the IX/EW process. Suspended solids can also become entrained in the organic used in the SX/EW process. This

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entrainment of suspended solids leads to formation of "crud" at the phase interface of the pregnant leach solution 18 and organic copper extractant solution. The crud, an emulsion of organic, pregnant leach solution 18, and suspended solids, adversely affects aqueous - organic phase separation and copper extraction efficiency. Carryover of entrained suspended solids and organic into the rich electrolyte stream causes contamination of the electrowinning process and affects copper product quality. Carryover of entrained suspended solids and associated organic into the lean leaching solution, "raffinate", results in the loss of large amounts of expensive organic.

The present invention specifically provides an improved method for copper recovery in which the suspended and colloidal solids are removed from the copper leach solution, leaving a cleaner pregnant leach solution 18 for copper extraction with organic lixivants or copper ion exchange resin beds. Accordingly, the invention represents an advance in the art of copper mining technology, as described in detail herein. Removal of colloidal solids from the PLS increases the copper loading efficiency onto the organic

lixiviant. Removal of colloidal solids from the PLS increases the copper/iron loading selectivity ratio of the organic lixiviant.

#### SUMMARY OF THE INVENTION

These and other needs are addressed by the metal recovery process of the present invention. The metal recovery process includes the steps of:

- (a) contacting a leach solution with a metal-containing material to form a pregnant leach solution containing at least a portion of the metal and suspended solids;
- (b) filtering the pregnant leach solution with a filter to form a permeate containing at least most of the metal in the pregnant leach solution and a concentrate containing at least most of the suspended solids in the pregnant leach solution; and
- (c) recovering the metal from the permeate.

A variety of metals can be recovered by this process. Most preferred metals are selected from the group consisting

of copper, nickel, zinc, cobalt, uranium and mixtures thereof.

The solids are relatively finely sized and therefore capable of being entrained in the pregnant leach solution. Commonly, the solids have a size of no more than about 1 micron and the colloids of no more than about .45 microns.

Filtration can be performed using a variety of microfiltration or ultrafiltration membranes. Preferably, the filter has a pore size ranging from about .003 microns to about .1 micron and more preferably from about .01 to about .05 microns.

Filtration is conducted such that most of the pregnant leach solution is contained in the permeate. The concentrate preferably comprises no more than about 20% of the pregnant leach solution and more preferably no more than about 5% of the pregnant leach solution. In contrast, the permeate preferably comprises at least about 80% of the pregnant leach solution and more preferably at least about 95% of the pregnant leach solution. In this manner, at least about 80% of the metal in the pregnant leach solution is contained in the permeate and no more than about 20% of



the metal in the pregnant leach solution is contained in the concentrate.

Filtration removes substantially all of the suspended solids and colloids from the permeate and places them in the concentrate. Preferably, the permeate is substantially free of suspended solids and more preferably comprises no more than about 1% of the suspended solids. In contrast, the concentrate preferably comprises at least about 95% and more preferably at least about 99% of the suspended solids in the pregnant leach solution.

A filter having a smaller filter size can further concentrate the metal in the permeate. The filter is preferably a nanofilter having a pore size ranging from about 8 to about 100 angstroms. Preferably, the concentration of the metal in the further concentrated permeate is at least about 150% and more preferably at least about 200% of the metal concentration in the permeate. Filtration is discussed in detail in U.S. Patents 5,116,511; 5,310,486; and 5,476,591, which are incorporated herein by reference fully in their entireties.

The concentrate can be recycled to step (a) for further leaching of the metal-containing material.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a flow schematic of a preferred embodiment according to the present invention.

#### DETAILED DESCRIPTION

The process improvements claimed in the present invention will result from utilizing a microfiltration or ultrafiltration membrane system to process the pregnant leach solution. As described above and shown in Figure 1, a strong leach solution 10, such as a sulfuric acid solution, passes downwardly through a heap or dump 14 of low-grade copper ore and a pregnant leach solution 18 is produced which contains remaining amounts of acid in combination with metal ions. In addition, the sulfuric acid, metal - ion pregnant leach solution 18 contains suspended solids and colloidal solids from the ore heap or dump 14. The pregnant leach solution 18 is collected from the bottom of the ore heap or dump 14, and passed through a membrane filtration

system 22 to remove suspended solids and colloids from the pregnant leach solution 18. The membrane system separates the pregnant leach solution 18 into two streams: permeate 26 and concentrate 30. The concentrate 30 stream includes substantially all of the suspended solids and colloidal solids in the pregnant leach solution 18. The permeate 26 is free of suspended solids and colloidal solids. However, sulfuric acid and metal ions in the pregnant leach solution 18 are not separated by the microfiltration or ultrafiltration membranes, and they remain equally in the concentrate and permeate streams.

The permeate 26 may be sent directly to the SX/EW plant 34 (or, alternatively, an IX/EW plant) for copper recovery. It may also be sent to a nanofiltration system (not shown) for copper ion concentration, followed by processing through the copper recovery plant 34.

The concentrate 30 may be sent to a settling pond (not shown) for deposition of the suspended solids, followed by processing through a nanofiltration membrane system (not shown), and, eventually, a copper recovery system. The concentrate stream may also be returned to the ore heap or

dump, where many of the suspended solids will be filtered out as the liquid works its way down through the heap.

Presently, most copper mining operations are not treating the pregnant leach solution to remove suspended solids. During periods of high suspended solids in the pregnant leach solution (such as rain or storm events), the SX/EW or copper recovery plants are shut down until the event is over and the suspended solids return to normal levels. The cost of shutting down during a storm event (lost production) was estimated at \$1.5 million dollars per day at one copper mining facility. With five to ten storm events per year in even dry or desert climates, the ability to filter out suspended solids and avoid process shut-downs has significant economic value.

Many copper mines eventually try to remove accumulated suspended solids by treating the rich electrolyte. At this point, the suspended solids have formed an emulsion called "crud", a mixture of suspended solids, organic, and electrolyte. The collected "crud" may be processed by decantation, centrifuging, or coarse filtration in an attempt to recover the expensive organic and remove the



membrane elements. The system would split the feed flow into 900 gpm of permeate and 100 gpm of concentrate. The concentrate would be sent to a settling pond, with an overflow to the SX/EW system, or returned to the ore dumps. The permeate, containing no suspended and colloidal solids, would be sent directly to the SX/EW plant, or processed through a nanofiltration membrane system and then sent to the SX/EW plant.

What is claimed is:

1. A method for recovering metal from a metal-containing material, comprising:

(a) contacting a leach solution with the metal-containing material to form a pregnant leach solution containing at least a portion of the metal and suspended solids;

(b) filtering the pregnant leach solution with an ultra- or microfiltration to form a permeate containing at least most of the metal in the pregnant leach solution and a concentrate containing at least most of the suspended solids in the pregnant leach solution; and

(c) recovering the metal from the permeate.

2. The method of Claim 1, wherein the metal is selected from the group consisting of copper, nickel, zinc, uranium and mixtures thereof.

3. The method of Claim 1, wherein the nanofilter has a pore size ranging from about .003 to about .1 microns.

4. The method of Claim 1, wherein the concentrate comprises no more than about 20% of the pregnant leach solution.

5. The method of Claim 1, wherein the permeate comprises at least about 80% of the pregnant leach solution.

6. The method of Claim 1, wherein the permeate is substantially free of suspended solids.

7. The method of Claim 1, wherein the permeate comprises no more than about 10% of the suspended solids contained in the PLS.

8. The method of Claim 1, wherein the concentrate comprises at least about 90% of the suspended solids in the pregnant leach solution.

9. The method of Claim 1, wherein the permeate comprises at least about 80% of the metal in the pregnant leach solution.



10. The method of Claim 1, wherein the recovering step comprises filtering the permeate with an ultra- or microfiltration to form a filtered permeate comprising at least most of the metal in the permeate.

11. The method of Claim 1, further comprising:

(d) contacting the concentrate with the metal-containing material.

12. The method of Claim 1, further comprising:

(d) filtering the permeate using a nanofilter to form a filtered concentrate containing at least most of the metal in the concentrate.

## ABSTRACT

A treatment system for removing suspended and colloidal solids from a copper leaching solution. A suspended solids, colloidal solids, and metal-ion containing liquid, i.e. "pregnant leach solution 18" from a copper leaching process, is passed through a microfiltration or ultrafiltration membrane system to produce a concentrate rich in suspended and colloidal solids and a permeate free of suspended solids and colloidal solids. The permeate may be sent directly to a copper recovery process for enhanced recovery of copper, or the permeate may be sent to a nanofiltration membrane process for copper concentration. The concentrate is returned to the top of the ore heap or dump for additional leaching, or it is sent to a settling pond for deposition of the suspended solids. This process removes suspended and colloidal solids from the copper leaching solution before they can enter the copper recovery plant. The result of this membrane filtration of the copper leaching solution is enhanced copper recovery with lower operating costs.

VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS  
(37 CFR 1.27(d) and 1.27(c)) - SMALL BUSINESS CONCERN

I hereby declare that I am an official empowered to act on behalf of the small business concern, HW PROCESS TECHNOLOGIES, INC. of 1201 Quail Street, Lakewood, Colorado 80215.

I hereby declare that the above-identified small business concern qualifies as a small business concern as defined in 13 CFR 121.3-18, and reproduced in 37 CFR 1.9(d), for purposes of paying reduced fees under section 41(a) and (b) of Title 35, United States Code, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons. For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has the power to control both.

I hereby declare that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to the invention, entitled "METHOD OF REMOVING SUSPENDED AND COLLOIDAL SOLIDS FROM COPPER LEACHING SOLUTION" and identified as Attorney File No. 3376-19PROV, described in application Serial No. to be assigned, filed concurrently herewith.

If the rights held by the above-identified small business concern are not exclusive, each individual, concern or organization having rights to the invention is listed below\* and no rights to the invention are held by any person, other than the inventor, who could not qualify as a small business concern under 37 CFR 1.9(c) or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d) or a nonprofit organization under 37 CFR 1.9(c).

\*NOTE: Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)

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Date: 10/24/97

By: [Signature]  
Harold W. Whatley  
Vice President of Finance and Administration



UNITED STATES DEPARTMENT OF COMMERCE  
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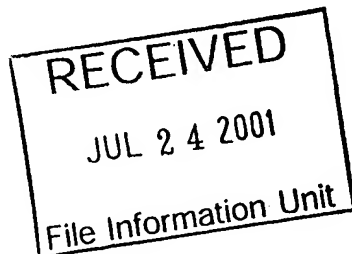
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# REQUEST FOR ACCESS OF ABANDONED APPLICATION UNDER 37 CFR 1.14(a)



In re Application of

Application Number

Filed

Group Art Unit

Examiner

Paper No. 2

Assistant Commissioner for Patents  
Washington, DC 20231

I hereby request access under 37 CFR 1.14(a)(3)(iv) to the application file record of the above-identified ABANDONED application, which is: (CHECK ONE)

- ☒ (A) referred to in United States Patent Number 6,152,186 column \_\_\_\_\_
- ☐ (B) referred to in an application that is open to public inspection as set forth in 37 CFR 1.11, i.e., Application No. \_\_\_\_\_ filed \_\_\_\_\_ on page \_\_\_\_\_ of paper number \_\_\_\_\_
- ☐ (C) an application that claims the benefit of the filing date of an application that is open to public inspection, i.e., Application No. \_\_\_\_\_ filed \_\_\_\_\_ or
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